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ELECTRICAL RELAXATION IN POLY(ETHYLENE OXIDE)(U) NAVAL
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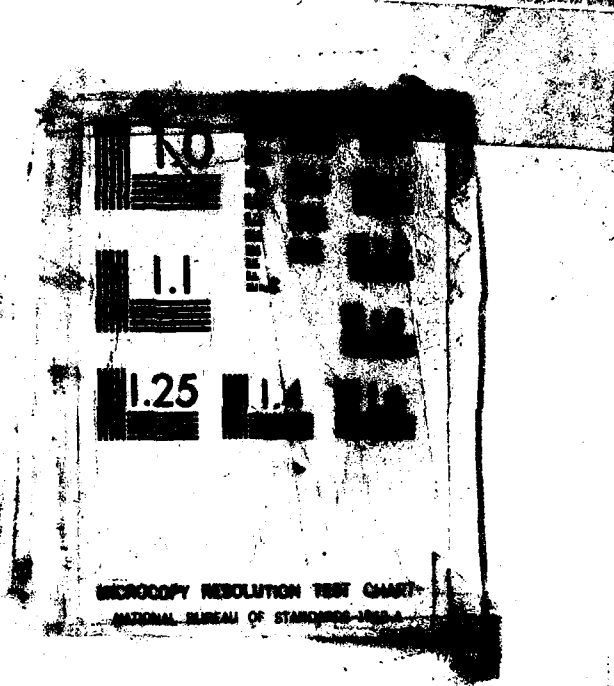
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ELECTRICAL RELAXATION IN POLY(ETHYLENE OXIDE)

by

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ELECTRICAL RELAXATION IN POLY(ETHYLENE OXIDE)

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ABSTRACT

Radio frequency complex admittance (DR) has been used to investigate electrical relaxation in poly(ethylene oxide) (PEO). The complex admittance measurements were performed at seventeen frequencies from 10 to 100,000 Hz in approximately equal logarithmic intervals using a fully automated dielectric spectrometer and measurements were performed in vacuum over the temperature range 5.5-350K. Two relaxations are observed at low temperatures which is consistent with the literature. In order to gain new information concerning the gamma relaxation, measurements were made at -94, -91, and -88°C at pressures up to 0.2 GPa. The shift in the peak frequency with pressure and hence the activation volume are found to be extremely small. This result combined with other work implies that the relaxation is associated with the motion of very small segments of the polymer chain. Originator
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I. INTRODUCTION

In previous papers [1-6], the authors have presented the results of audio frequency electrical relaxation (DR) measurements on "pure" PEO and PEO complexed with a variety of alkali metal and alkaline earth salts. In addition, thermally stimulated depolarization measurements have recently been reported [7]. In general, three relaxations are observed. The two lower temperature relaxations, α_g and γ , are those which have been most widely studied [1-15]. α_g is associated with the glass transition as was shown in a very early investigation [10]. At least four explanations of the γ relaxation have been suggested [15-20], the most likely of which are chain end hydroxyl groups [16] or a gauche minus-gauche plus transition [14]. In order to gain further information concerning the γ relaxation, high pressure measurements were carried out and the results are reported in the present paper.

II. EXPERIMENT

The polymer was obtained from Polysciences, MW 5×10^6 or 4×10^6 . Some samples were prepared by dissolving the PEO in methanol at about 60°C . The resulting solutions were poured onto a teflon plate and allowed to dry in air. After completing the drying under vacuum at 60°C for 24 hours, aluminum electrodes were evaporated onto the resulting films. These films were then cut to form disks about 8 mm in diameter and 0.7 mm thick. Other "pure" samples were pressed at about 100°C using a Buehler Simplimet II press. Three terminal measurements were performed on the hot-pressed

samples which were approximately 25 mm in diameter and 1.5 mm thick. No difference between the hot-pressed samples and the cast films was noted other than small differences in the apparent real part of the dielectric constant which was attributable to two terminal vs. three terminal measurements.

The high pressure measurements were carried out in the vessel described elsewhere [21]. The pressure fluid was a mixture of pentane and Spinesstic 22 and the temperatures were achieved using a freon bath cooled by gas from liquid nitrogen circulating through copper coils. The vacuum measurements were performed in the dewar described elsewhere [2] and the audio frequency complex impedance measurements were performed using a fully automated bridge constructed by one of the authors (C.G.A.) which operates at seventeen frequencies over the range $10\text{--}10^5$ Hz.

III. RESULTS AND DISCUSSION

Typical results for the temperature dependence of the imaginary part of the dielectric constant are shown in Fig. 1. The data are replotted in Fig. 2 as a "conductivity" plot. The DC conductivity at room temperature is on the order of 10^{-9} (ohm-cm) $^{-1}$. This value is rather high and is attributed to the presence of trace impurities in the sample.

Two strong peaks dominate the spectrum. The higher temperature peak, α_g , occurs at about 223K and 10^3 Hz and is associated with the glass transition [10]. This relaxation is interesting in that for most samples, a peak is never observed in the ϵ'' vs frequency plot at any single temperature in the frequency range 10 to 10^5

Hz, though it is observed in a temperature sweep as is obvious from Fig. 1. Further characteristics of this relaxation along with its pressure dependence will be given elsewhere [1].

The second strong feature, the γ relaxation has a peak at about 175K and 1000 Hz in an ϵ'' vs. T(K) plot. It is very broad as shown by the Cole-Cole plot in Fig. 3. In fact, the Cole-Cole parameter is about 0.69. However, the relaxation is slightly asymmetric and to properly treat the relaxation requires a more complex distribution of relaxation times. To analyze the vacuum data, the imaginary part of the two parameter Havriliak-Negami distribution was used [22]:

$$\epsilon'' = \frac{\epsilon_s - \epsilon_\infty}{[1 + (\omega/\omega_p)^{(1-\alpha)]^\beta} \quad (1)$$

This expression was fit to the data using a least squares "reduced plot" technique. First, an arbitrary peak position and peak height and approximate values of α and β were chosen. Second, the data at a few temperatures were shifted to the vicinity of the arbitrary peak, the shifts being adjusted to give a "best fit" to the distribution. Third, the parameters were varied to "best fit" to the data. The second and third steps were repeated until all of the data had been treated. The results are shown in Fig. 4 where the data and best fit curve are plotted. The significant Havriliak-Negami parameters are $\alpha=0.567$ and $\beta=0.390$. It is obvious that the Havriliak-Negami distribution provides an excellent fit to the data.

The γ relaxation has been variously attributed to a "crankshaft mechanism" [17,18], "3-bond" or "4-bond" motions [19,20], chain end hydroxyl groups [16], or the $tg^+t \leftrightarrow tg^-t$ transition [14]. In order to gain further information concerning the γ relaxation, high pressure measurements have been carried out. Typical data are shown in Fig. 6. In order to determine the peak positions, and hence the relaxation times, the imaginary part of the Cole-Cole distribution:

$$\epsilon'' = \frac{\epsilon_s - \epsilon_\infty}{[1 + (\omega/\omega_p)^{(1-\alpha)]} \quad (2)$$

was fitted to the data. As was discussed above, the Cole-Cole distribution is not sufficient to describe the complete relaxation. However, the objective of this part of the analysis is to measure the shift in peak position with pressure and the Cole-Cole distribution can be used for that purpose so long as the shifts are small and the fits are done in the same way for each pressure. Typical data and best fit curves are shown in

Fig. 5. The reciprocal of the peak position is plotted vs. pressure in Fig. 6 along with the best fit straight line. The slopes of the curves are tabulated in Table I for all of the data.

These data were used to calculate the activation volume via:

$$V^* = kT \frac{d \ln \tau}{dP} \quad (4)$$

and the results are listed in Table I. The activation volumes are extremely small. This result argues against multiple bond mechanisms such as the "crankshaft" or "4-bond" mechanisms. Rather, it provides support for either chain end hydroxyl groups or motion of very small segments of the polymer chain such as the $tg^+t \rightleftharpoons tg^-t$ mechanism.

In most of their previous papers on this subject, the authors have interpreted their data in terms of the latter reorientation mechanism. Although the authors cited preliminary, unpublished work in favor of chain end hydroxyl groups in two papers [2,6], that work is not complete at the present time. The authors have subsequently obtained some evidence against chain end hydroxyl groups [1]. Consequently, at the present time, the evidence favors assigning the γ relaxation to very small segments of the polymer chain.

V. SUMMARY

In summary, then, it is shown that the γ relaxation is assymetric and thus not well described by a one parameter distribution. A very good fit to the data was obtained using the two parameter Havriliak-Negami

distribution. In addition, data on the pressure dependence of the γ relaxation peak are presented. Using a Cole-Cole fit to the data, the shifts in peak position as a function of pressure are evaluated and used to calculate the activation volume for the reorientation process. The small value for the activation volume argues against the mechanisms proposed to explain the γ relaxation in terms of motion of large segments of the polymer chain, such as the "4-bond" or "crankshaft" mechanisms. The data do not allow a distinction to be drawn between the " $tg^+t \nleftrightarrow tg^-t$ " and the "chain end hydroxyl group" models, however, other work favors the former mechanism.

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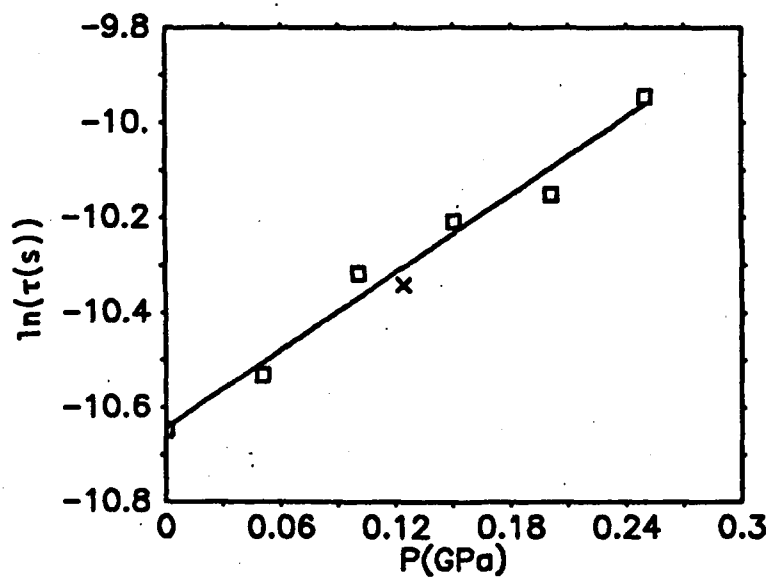
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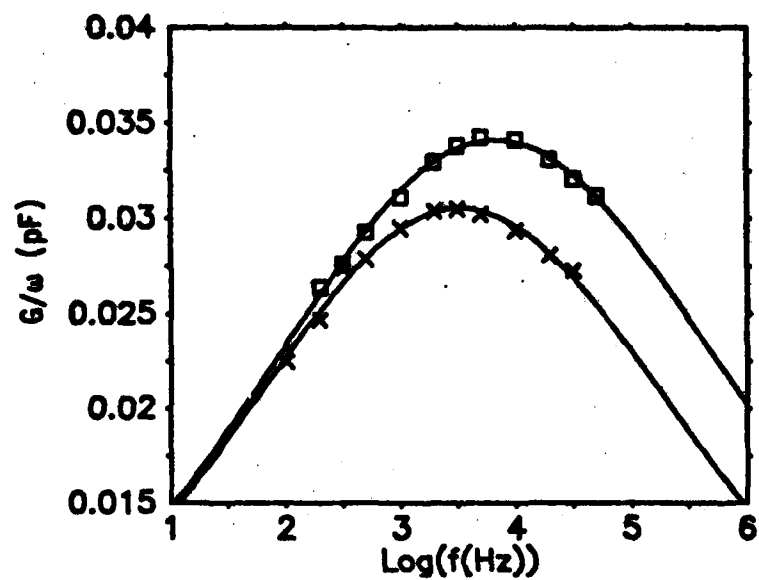
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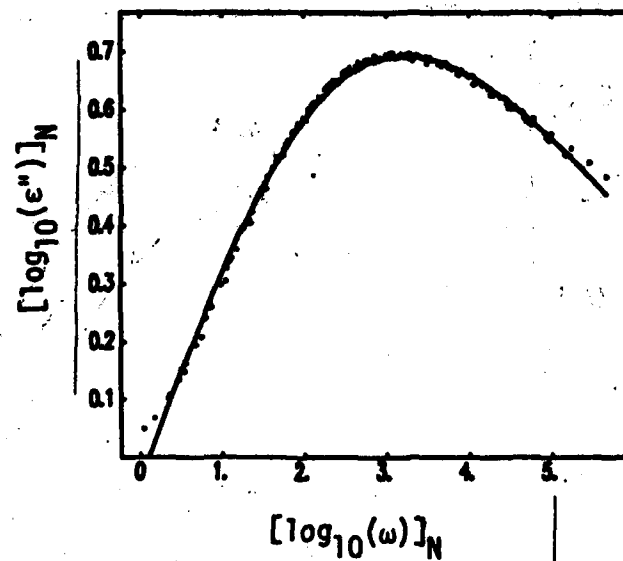
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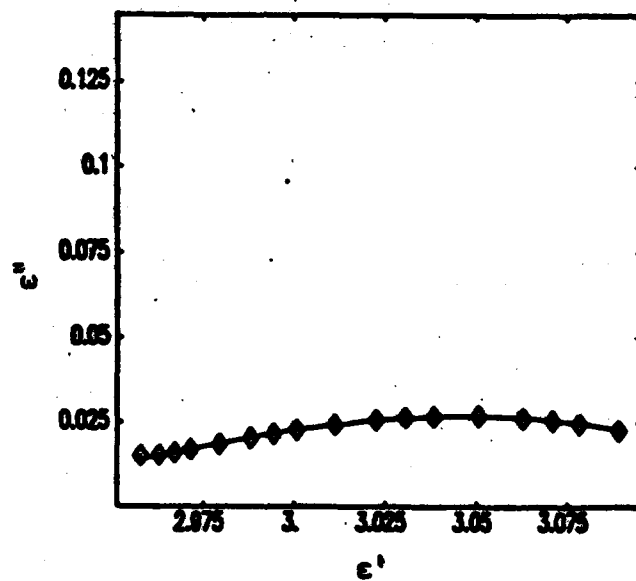
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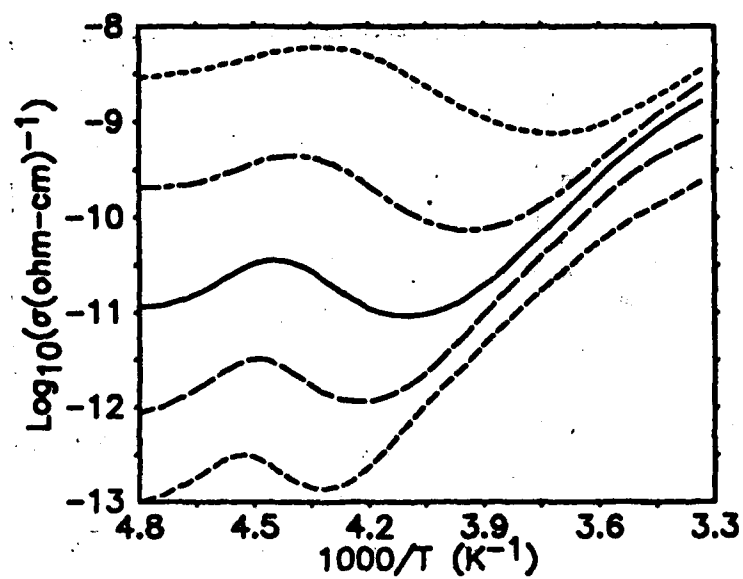
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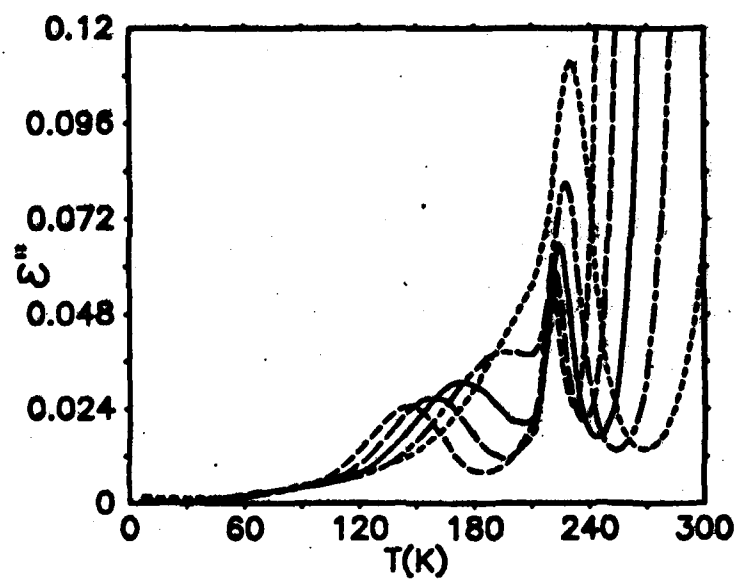


Conte et al. Fig. 4



Tantarella et al Fig 3





E. tenall et al. Fig. 1

FIGURE CAPTIONS

Figure 1. ϵ'' vs. $T(K)$ for pure PEO. The curves (from left to right) are: medium dash- 10 Hz; long dash- 10^2 Hz; solid- 10^3 Hz; chain link- 10^4 Hz; short dash- 10^5 Hz. Straight line segments connect the datum points which are not shown.

Figure 2. $\ln(\sigma(\text{ohm-cm})^{-1})$ vs. $1000/T$ (K^{-1}) for pure PEO. The curves are: medium dash- 10 Hz; long dash- 10^2 Hz; solid- 10^3 Hz; chain link- 10^4 Hz; short dash- 10^5 Hz. Straight line segments connect the datum points which are not shown.

Figure 3. Cole-Cole plot for the gamma relaxation in pure PEO at 170K. Also shown is the best fit Havriliak-Negami curve.

Figure 4. "Reduced plot" for the gamma relaxation in pure PEO. The data points and best fit Havriliak-Negami curve are shown. The peak position and strength are arbitrary.

Figure 5. Arrhenius plot of the peak shift for the results shown in Figure 4. Also shown is the best fit straight line.

Figure 5. G/ω (pF) vs. $\log_{10}(f(\text{Hz}))$ at 1 atm (squares) and 0.3 GPa (crosses) for the gamma relaxation in PEO at 182 K. Also shown are the best fit Cole-Cole curves.

Figure 6. $\ln(\tau(s))$ vs. $P(\text{GPa})$ for the gamma relaxation in pure PEO at 182 K. Also shown is the best fit straight line.

Table 1

Pressure derivative of the relaxation time and activation volume for the gamma relaxation at various temperatures for pure PEO.

T(K)	$\frac{d \ln \tau}{dP}$ (GPa) ⁻¹	ΔV^* (cm ³ /mol)
<u>Sample #1</u> (Hot pressed)		
182	3.0	4.5
185	3.4	5.3
<u>Sample #2</u> (Cast film)		
179	2.9	4.3
185	3.1	4.7

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